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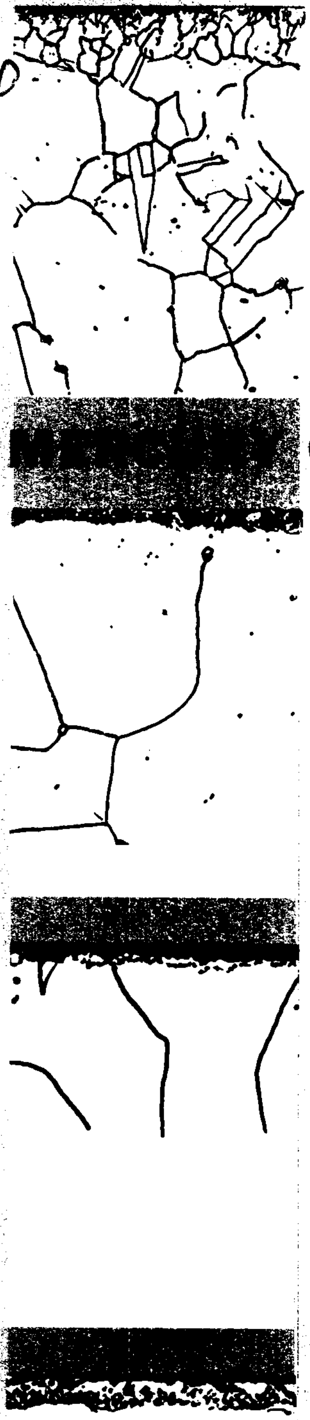

[illegible] CORROSION LOOP TESTING PROGRAM

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Report No. 0584-04-3 (Quarterly)

July 1962

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6 August 1962

Report No. 0584-04-3

MERCURY CORROSION LOOP TESTING PROGRAM

Contract NAS 3-1925

No. of Pages: 26

Period Covered: 1 April through 30 June 1962

AEROJET-GENERAL CORPORATION

Azusa, California

NASA CR-50,341

ABSTRACT 17290

This report describes the fabrication and testing accomplished during the third quarter (April - June 1962) of the Dynamic Mercury Corrosion Loop Testing Program. Operation of the first of ten loops to be tested was begun, and a second loop was fabricated. Tubing made of special alloys was procured for the fabrication of additional loops.

CONTENTS

CONTRACT FULFILLMENT STATEMENT

Work at Aerojet has been authorized under NASA Contract NAS 3-1925. Testing is being conducted at Aerojet-General Nucleonics (an Aerojet subsidiary), San Ramon, California, under Subcontract No. OP328862. This is the third in a series of quarterly reports submitted in partial fulfillment of Contract NAS 3-1925.

I. INTRODUCTION

Within the aerospace industry, several power conversion systems which employ mercury boiling and condensing cycles are under development. These systems share a common need for quantitative information about the long-term compatibility of mercury with mercury-containment materials.

The Mercury Corrosion Loop Testing Program, currently being conducted by the Aerojet-General Corporation, is designed to provide such information. The objective of the program is to produce sufficient experimental data to provide a firm basis for selection of a suitable mercury-containment material. The material must be able to withstand a nominal mercury boiling temperature of 1075°F for an operational life of 10,000 hours. Several alloys, including Haynes 25, will be tested in ten forced-convection test loops. Testing periods will exceed 2000 hours and, on some loops, will approach 10,000 hours. The loops will duplicate as closely as possible the cycle conditions and flow characteristics of the SNAP-8 power conversion system.

In addition, the loop data will be used to estimate the location and degree of corrosion attack that can be expected in the operational system.

Current objectives of the program are to complete a 2000-hour test run of the first Haynes 25 loop, to complete fabrication and assembly of three other Haynes 25 loops, to initiate fabrication of the unclad 9Cr-1Mo loop, and to evaluate the clad 9Cr-1Mo and columbium tubing.

II. SUMMARY OF ACCOMPLISHMENTS - 1 APRIL THROUGH 30 JUNE 1962

The first Haynes 25 corrosion test loop was checked out and operated intermittently, for a total running time of 380 hours.

The design of the second Haynes 25 boiler was revised. The boiler was then fabricated, along with a slightly modified radiator. The second loop is now being assembled.

Special alloy tubing of 9Cr-1Mo and AM 350 was received and inspected. A contract was awarded for fabrication of the 9Cr-1Mo boiler.*

The first billets of co-extruded 9Cr-1Mo tubing clad with Type 316 stainless steel were successfully extruded by the vendor.

III. DISCUSSION

A. SPECIAL ALLOY PROCUREMENT

A sufficient quantity of Haynes 25 tubing was obtained to permit fabrication of the four Haynes 25 test loops. For fabrication of the remaining six loops for the test plan (discussed in the previous quarterly report), special alloy tubing must be procured.

Procurement was initiated for welded and redrawn AM 350 tubing, unclad 9Cr-1Mo, 9Cr-1Mo clad with Type 316, and columbium clad with Type 316. The first two alloys were received and inspected by dye check and ultrasonic inspection. The other two alloys are in process, with delivery expected shortly.

The twisted tape stock of AM 350, 9Cr-1Mo, and columbium for the boiler vortex generator was received. Bar stock of 9Cr-1Mo for the centrifugal pump housings was ordered.

A survey was made of possible vendors of seamless Hastelloy-N tubing for the boiler heater sheaths, but long delivery times quoted (i.e., 7 to 9 months) eliminated any consideration of this alloy. Inconel will be used instead. Hastelloy-N plate was procured and will be used for the internal supports of the boiler.

B. ALLOY EVALUATION

First extrusions of the 9Cr-1Mo tubing clad with Type 316 were made by the vendor during the quarter. The extrusions were approximately 1.0 in. OD and will be cold-drawn to final size. Samples of the clad tubing, as extruded, were received and a section of the tubing was heated to 1400°F in air through the following cycle:

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*The following abbreviations are used in this report:

Type 316 = Type 316 stainless steel
9Cr-1Mo = 9 chromium - 1 molybdenum steel alloy
AM 350 = AM 350 precipitation-hardening stainless steel

1. Heat furnace to 400°F
2. Place sample in furnace and heat to 1400°F in increments of 200°F/hour
3. Hold sample at 1400°F for 1 hour
4. Cool sample to 1000°F in decrements of 200°F/hour
5. Cut power and allow sample to cool in furnace.

The bond between the Type 316 cladding and the 9Cr-1Mo base was examined before and after the heat treatment described above. The bond was not affected and no separation was found (Figure 1).

Stress-rupture tests are being conducted on AM 350 at 1300°F in air to determine the stress-rupture strength of the alloy. No published data on stress-rupture strength could be found for solution-annealed AM 350 at temperatures near 1300°F.

Stress-rupture tests completed to date are summarized in Table 1. The results of these tests, as plotted in Figure 2, indicate that solution-annealed AM 350 exposed to temperatures between 1025°F and 1100°F and then cooled to room temperature has a lower stress-rupture strength than normal solution-annealed AM 350. Extrapolation of the stress-rupture data obtained at 1300°F indicates that the maximum stress in the boiler tube in the loop should not exceed 4000 psi if the AM 350 loops are to be operated for extended periods of time.

Evaluation of the mercury pump from Loop No. 1 was started. The evaluation will study the corrosion products and the mass-transfer deposits found in the pump. The reason for the failure of the pump will also be investigated.

C. LOOP FABRICATION

The principal task during this quarter was to operate the first Haynes 25 corrosion loop and check out additional Haynes 25 centrifugal pumps.

Problems encountered with the first loop resulted in several changes being made in the design of the second loop. These changes are outlined below.

1. Welding cracks in the first radiator assembly were attributed to the heat cycle utilized in the copper fin brazing operation. New tubing and a revised fin braze were used to make the radiator tubes for Loops 2, 3, and 4.

2. In addition to the plug insert (discussed in the previous quarterly report), the boiler coil was modified so as to extend out of the bottom head. Thus, the boiling will occur while the mercury flows upward.

3. The loop pressurizer was relocated on the pump suction side; the mercury level will vary only 1 ft during operation.

4. The turbine simulator was modified to operate at 840 lb/hour with sonic flow in one nozzle. The expansion heat will be removed by conduction through a copper fin brazed to the 3/4-in. Haynes 25 tube. Figure 3 shows the new design.

A special blade erosion test will operate in parallel with the turbine simulator. A flow rate of 360 lb/hour will pass through the blade row shown in Figure 4.

The test facility was completed early in the quarter. A larger-capacity air compressor was procured and installed to supply instrument air.

The Haynes 25 condenser for Loop No. 2 was completed and leak-tested. The third condenser is being leak-tested. The fourth condenser is ready for welding.

The second and third Haynes 25 boilers have been completed and leak-tested. A fourth boiler is partially completed. A delay was experienced because of the presence of edge cracks in the top head. These cracks were removed by machining 1/4 in. of stock from the edge. Inspection with dye penetrant showed no additional cracks present.

The Haynes 25 boiler coil for the fourth boiler assembly is shown in Figure 5.

Three Haynes 25 centrifugal pumps were completed and checked out. One pump is being reworked.

The two electrodynamic pumps originally scheduled for shipment in April were rescheduled for shipment in August. Vendor delays made such action necessary.

Assembly of the second Haynes 25 loop was initiated after revisions in the loop piping and changes in the design of the turbine simulator.

D. LOOP OPERATION

Operation of the first Haynes 25 loop began on 25 April and continued intermittently through June. Table 2 is a summary of the operation. A total of 380 hours operating time has been logged. Shutdowns were caused by mechanical failures, or by loop revisions to improve flow control. The characteristics of the loop are discussed below together with the problems encountered in operation.

The boiler coil in this loop was designed to obtain 150°F of superheat at 1200 lb/hour. Since the inside surface area is nearly 10 sq ft, an average heat flux between 15,000 and 16,000 Btu/hour-sq ft is required. It was anticipated that the coil would perform like the SNAP-8 boiler design, wherein a heat flux of 30,000 Btu/hour-sq ft would occur up to 50% quality. From 50% quality, the heat flux would diminish to 5000 Btu/hour-sq ft at saturation. A boiling heat flux of 30,000 Btu/hour-sq ft is typical of the values obtained where wetting occurred on the SNAP-8 program. Initial operation of the loop showed a heat flux of 5000 Btu/hour-sq ft, which is identical to the film boiling values obtained with Haynes 25 and the SNAP-8 program.

During run No. 6, a gradual improvement in heat flux was noted up to a value of 8000 Btu/hour-sq ft. However, the tube wall temperature was 1200°F and film boiling occurred. The heat transfer characteristics of the loop were investigated by varying the mercury conditions, flow rate, and NaK temperature distribution. Various NaK-to-mercury temperature differences were tried, ranging from 25°F to 200°F. The operational modes tried are as follows:

1. A reduced mercury flow of 500 lb/hour at 275 psia discharge pressure and 1075°F saturation temperature.

2. A reduced flow rate of 800 lb/hour at 130 psia discharge pressure. Saturation temperature was approximately 900°F. Various amounts of superheat were obtained at this condition, according to the heat balance and temperature measurements.

3. Increased flow rate of 1600 lb/hr at 275 psia discharge pressure with a saturation temperature of 1075°F. The exit quality was 50% and the average heat flux was 8,000 to 10,000 Btu/hr-sq ft.

It was concluded that the boiler coil does not contain enough heat-transfer surface for the flow rate of 1200 lb/hour. Two concurrent factors are responsible for the deficiency: (1) Low velocity occurs in the entrance region where heat flux depends upon velocity, and (2) mercury appears to wet Haynes 25 with difficulty, so that low boiling fluxes typical of film boiling are obtained. Since the twisted tape is of only a moderate twist, the vortex flow does not augment the film type heat fluxes enough to transfer the required heat.

The second Haynes 25 loop will utilize a plug insert to correct the low velocity condition, but little can be accomplished with respect to low boiling fluxes unless a different cleaning technique or treatment will mercury-wet the Haynes 25. It is anticipated that a more rapid wetting action on the 9Cr-1Mo alloy will occur. Consideration is being given to connecting two boilers in series, to obtain superheated vapor at the design flow rate and pressure level.

In addition to the reduced heat-transfer performance in the boiler, several other operational difficulties were encountered. Flow fluctuations occurred, caused by an unstable pneumatic control system. This was corrected by installing a positioner on the pneumatic control valve and by installing a dampener in the signal line from the differential pressure cell to the flow recorder-controller.

The second difficulty involved the turbine simulator trim valve, which is limited to 1000°F operation. Because the boiler was not producing design conditions, insufficient throttling occurred in the turbine simulator nozzles. To obtain the 700°F condensing temperature required, the trim valve had to be exposed to 1000°F vapor in the throttling process. The valve spring became annealed during run No. 3, so copper cooling coils were wrapped around the valve body to correct the situation.

The most troublesome part of the loop has been the centrifugal pump, which exhibits poor net positive suction head characteristics. Also, when the pump is operated nearly throttled, the front vanes generate more head than the back vanes and the seal cavity is flooded. Two bearing failures have resulted from mercury entering the top assembly.

The bearing failures reported during the last period with the first Type 316 pump were found to be a result of the impeller rubbing on the housing. It was concluded that the bearing design is adequate to withstand the thrust and radial loads, provided that the mercury can be prevented from reaching the bearing assembly.

Since the present pump impeller develops 500 psi discharge pressure, it was decided to trim the diameter of the front vanes to reduce the reverse pumping effect. The trimmed impeller developed 410 psi discharge pressure and the reverse pumping was reduced. However, the suction-head requirements are now 8 to 9 ft instead of 1 to 2 ft for the original blade diameter.

Two design changes are to be made on three Haynes 25 pumps: the number of back vanes will be increased from four to eight, and a jet pump will be used on the suction to reduce cavitation.

The Haynes 25 pump which had been used for test runs 1 through 6 was removed from the loop for inspection. Figure 6 is a photograph of the impeller. The black, powdery deposit shown on the impeller was analyzed by X-ray fluorescence and found to consist of nickel, chromium, iron, and cobalt. The Metallurgy Group is completing an analysis of the deposits and other particulate matter present in the residual mercury.

IV. WORK PLANNED FOR THE NEXT QUARTER

Operation of the first Haynes 25 loop will continue toward the 2000-hour objective.

The second Haynes 25 loop will be assembled and testing will begin.

The 9Cr-1Mo tubing clad with Type 316 and columbium tubing will be received and evaluated. If the materials prove acceptable, boiler fabrication will be initiated.

Fabrication of the unclad 9Cr-1Mo boiler, condenser, pumps, and valves will be completed and the loop will be assembled.

TABLE 1

RESULTS OF STRESS-RUPTURE TESTS ON SOLUTION ANNEALED AM 350 STAINLESS STEEL*

Pre-test Exposure				
Time, hour	Temperature, °F	Stress, psi	Average Time to Rupture, hours	Number of Tests
Unexposed		15,000	60.0	1
Unexposed		10,000	560.0	2
Unexposed		8,000	1455.0	2**
Unexposed		8,000	Test not completed	2
2000	1100	20,000	3.4	1
2000	1100	15,000	14.3	1
2000	1100	10,000	126.0	1
2000	1025	10,000	101.0	1
2000	1025	8,000	485.0	1

*All tests made at 1300°F in air

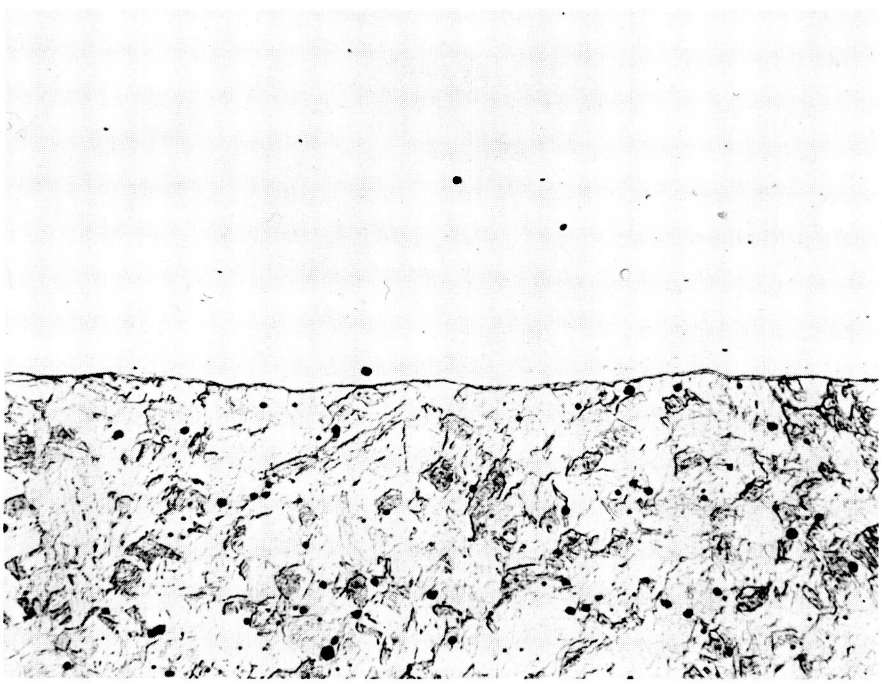
**Electric power failed after 1400 hours of the test. The test specimens were cooled to room temperature before the test was resumed.

TABLE 2

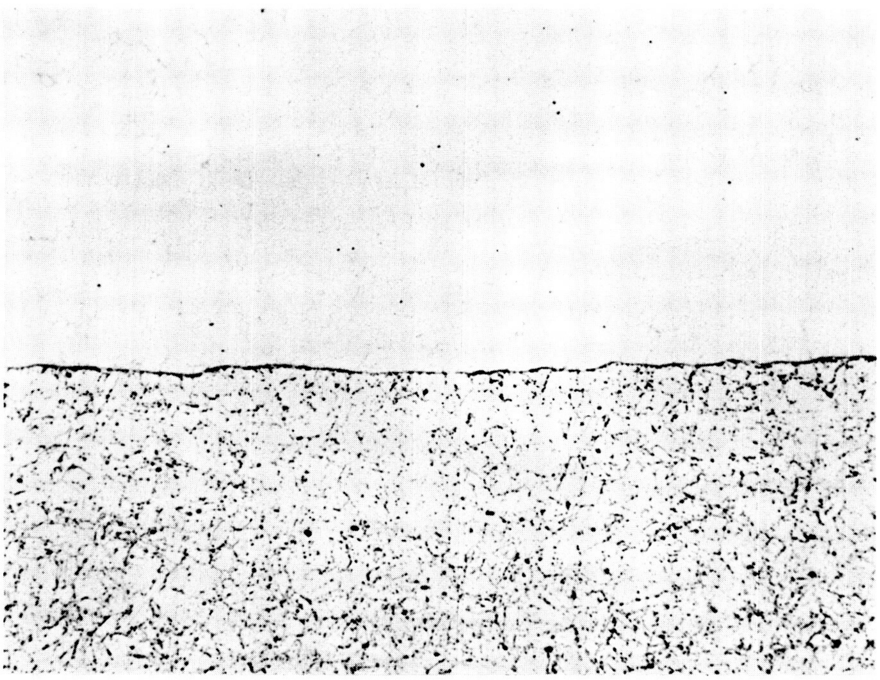
OPERATION DATA, LOOP NO. 1 - HAYNES 25

Run No.	Start	Duration, hours	Approximate Flow Rate, lb/hour	Boiler Outlet Pressure, psig	Vapor Quality, %	Remarks
1	25 April	5	1200	260	40	Rupture of fitting on argon high-pressure manifold caused shutdown. Instability of flow control system noted.
During interim period:			Installed new flow control valve, manual by-pass valve around flow control valve, and one 2.5 kw tubular electric heater to the boiler feed line.			
2	11 May	76	1200	260	40	Flow control still unstable. Run stopped to install positioner on flow control valve.
During interim period:			Installed positioner on flow control valve and two additional 2.5 kw tubular electric heaters to the boiler feed line.			
3	23 May	45.5	1200	260	40 - 50	Flow control still unstable. Turbine simulator outlet valve failed. Turbine simulator cooling water outlet clogged.
During interim period:			Removed cooling water connections from turbine simulator, filled water jacket with 30 mesh aluminum powder, added copper tubing coil to outside of turbine simulator for water cooling. Added mercury by-pass line from pump cavity to pressurizer (as return).			
4	28 May	10	300	260		Turbine simulator cooling-water line clogged.
During interim period:			Installed distilled water cooling supply with intercooler to plant cooling-water supply to turbine simulator. Replaced destroyed preheater section of loop tubing (section overheated during heat loss calibration). Replaced heaters and cooling coils on turbine simulator valve.			
5	6 June	24	1200	260		Leak on NaK side of boiler
During interim period:			Welded cap over leak in boiler and installed variable restrictor in transmission line from flow D/P cell.			
6	8 June	220	Varied		50	Heat flux 8,000 - 10,000 Btu/hour-sq ft. Pump bearings and seal failed. Loop shut down for pump replacement.
			500	260		
			800	115		
			1600	260		

PHOTOMICROGRAPHS OF THE 9Cr-1Mo TUBING CLAD WITH 316 STAINLESS STEEL



500X AS EXTRUDED ETCHED 2204



500X AFTER HEAT CYCLING TO 1400°F 2203
ETCHED

Figure 1

AM-350 STRESS - RUPTURE TESTS AT 1300°F

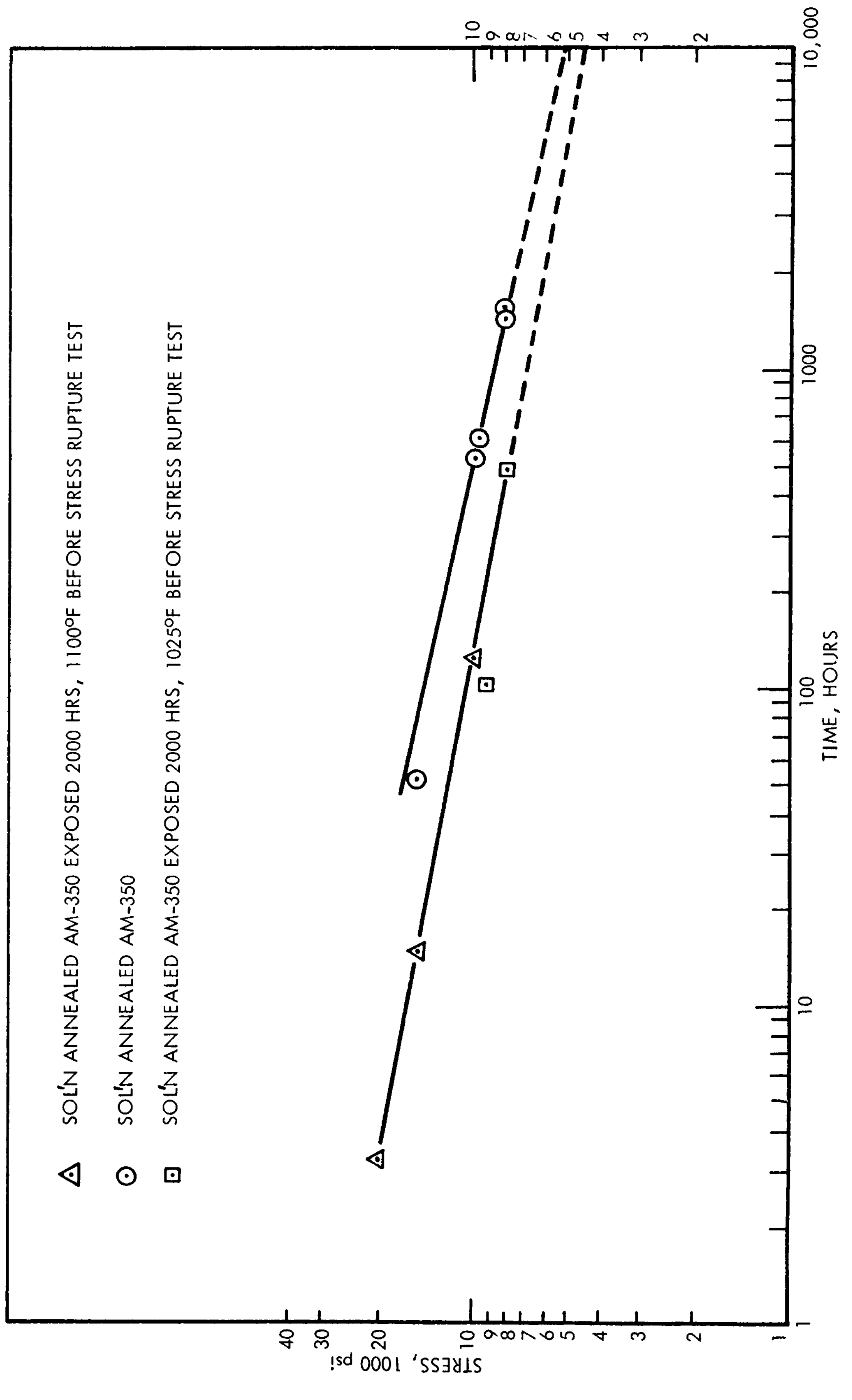
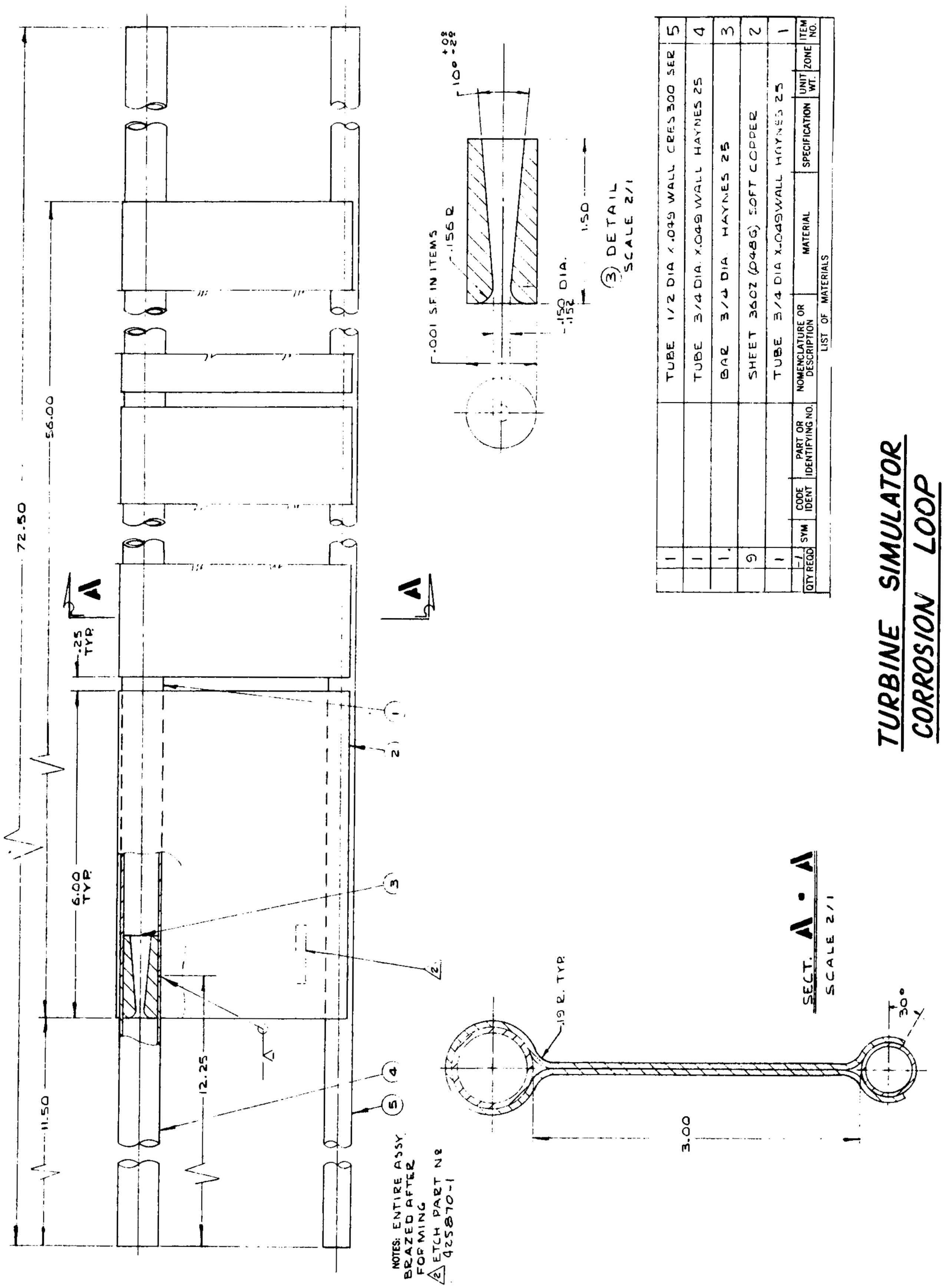


Figure 2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56.00
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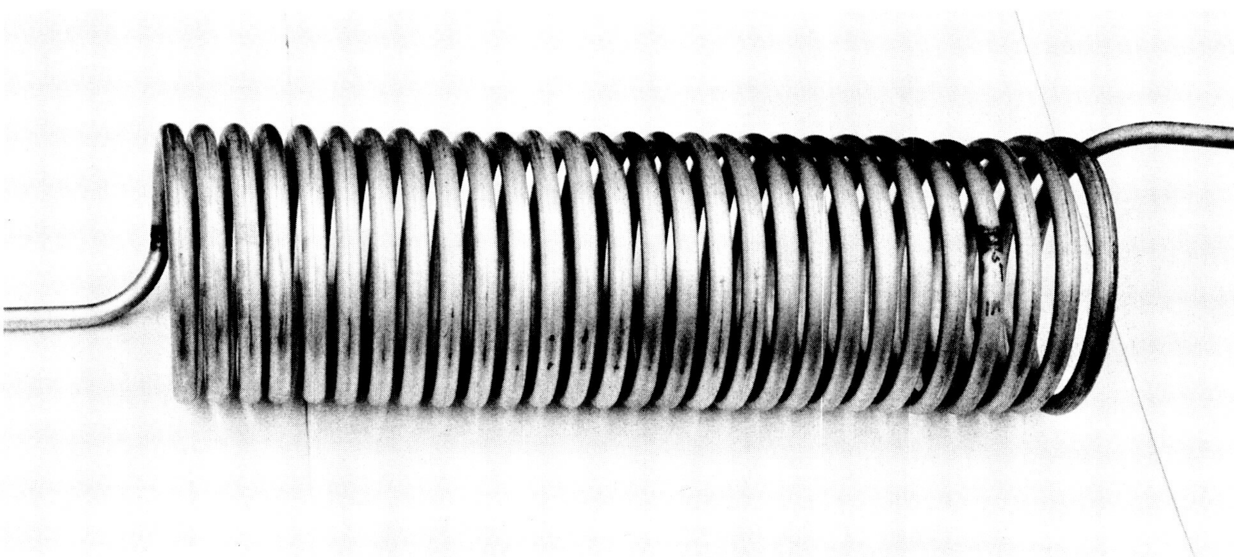
NOTES: ENTIRE ASSY BRAZED AFTER FORMING
2 ETCH PART No 425870-1
TUBE 1/2 DIA x .049 WALL CRES 300 SER 5
TUBE 3/4 DIA. x .049 WALL HAYNES 25 4
BAR 3/4 DIA HAYNES 25 3
SHEET 36OZ (048G) SOFT COPPER 2
TUBE 3/4 DIA x .049 WALL HAYNES 25 1
QTY REQD
SYM
CODE IDENT
PART OR IDENTIFYING NO.
NOMENCLATURE OR DESCRIPTION
MATERIAL
SPECIFICATION
UNIT WT.
ZONE
ITEM NO.
LIST OF MATERIALS
TURBINE SIMULATOR CORROSION LOOP

Assembly for Blade Erosion Test, With Detail of Blade Row

Figure 4



HAYNES 25 BOILER COIL

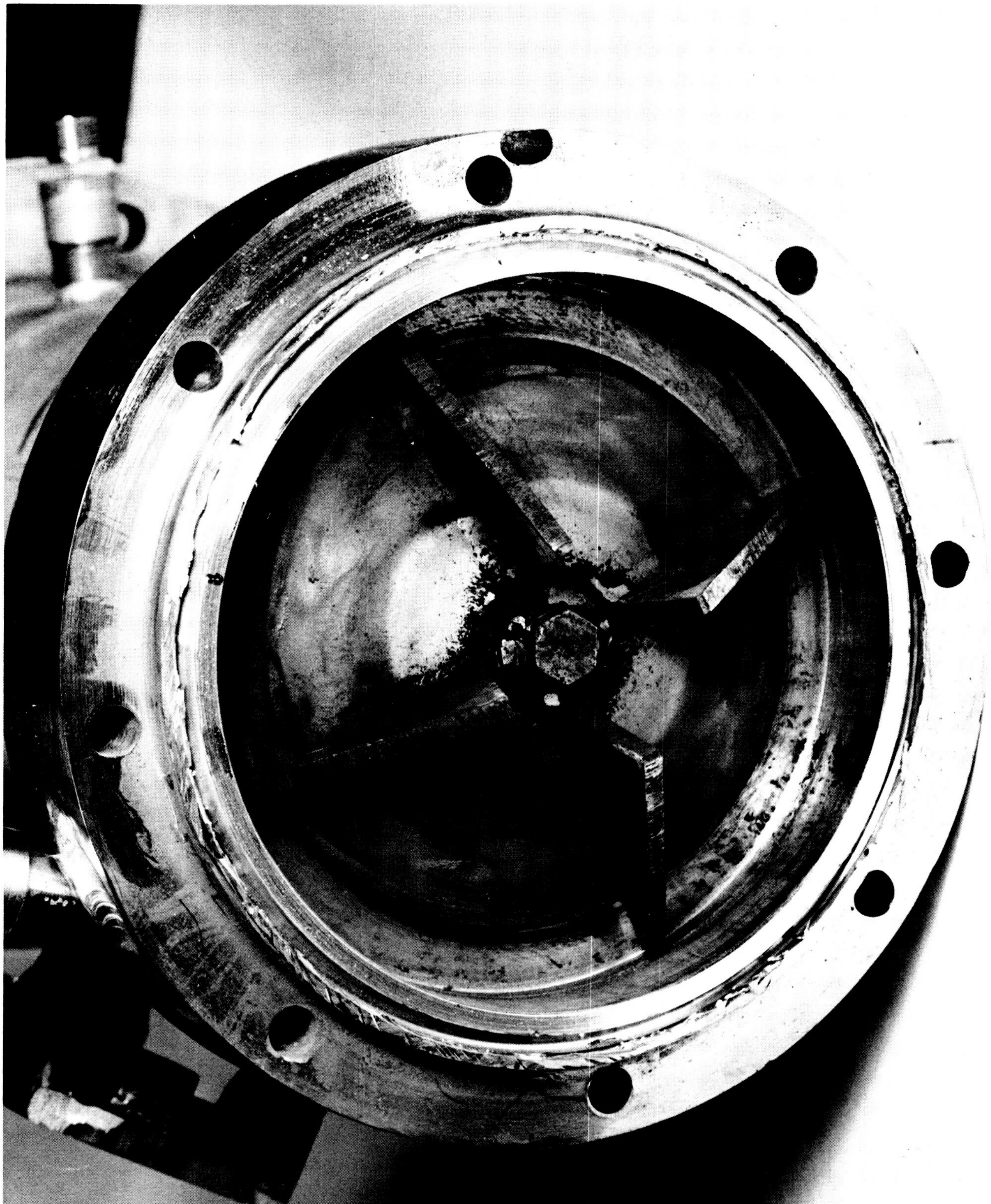


Figure 6

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